Elimination of Oil From Exhaust Steam

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ELIMINATION OF OIL FROM EXHAUST STEAM

A THESIS

PRESENTED BY

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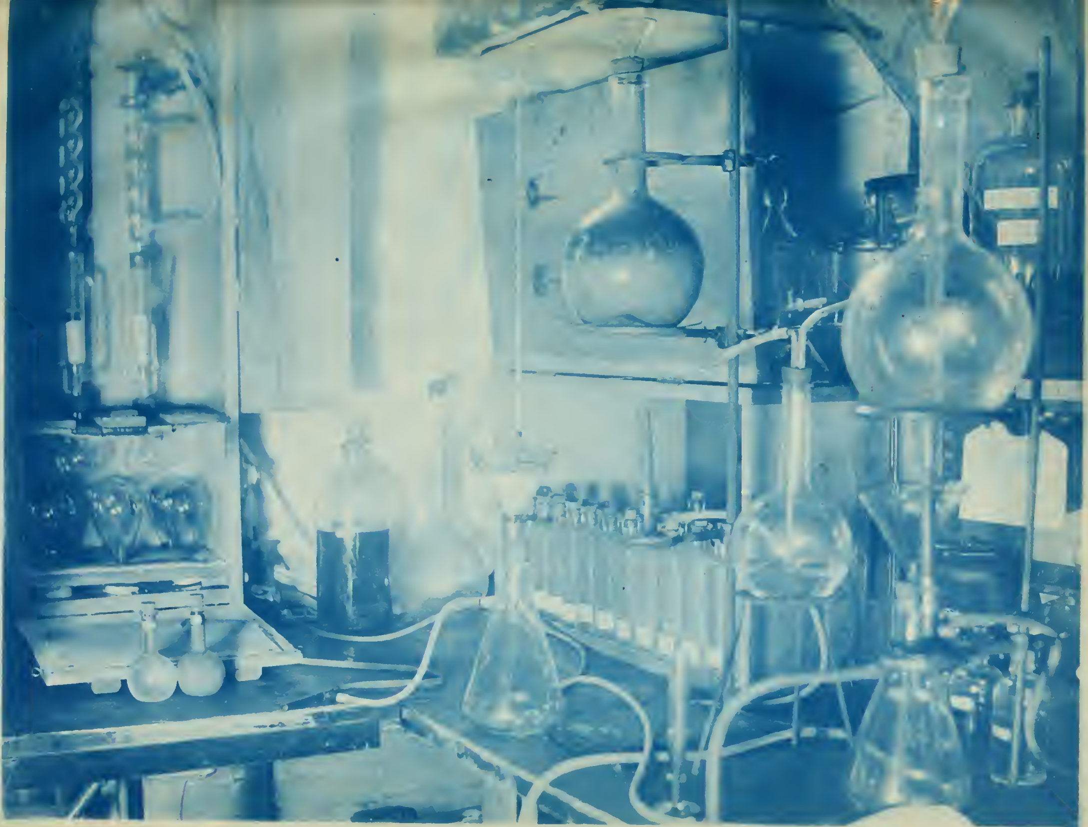

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OBJECT

The object of this investigation was to determine the best and most practical method of determining the percentage of oil existing in the exhaust steam from an engine.

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The Elimination of Oil from Exhaust Steam.

Oil is eliminated from exhaust steam by oil separators, or, as they are often called, eliminators or grease extractors, before it passes to the condenser or heating system.

When live steam enters an engine it carries with it oil necessary to lubricate the engine. The quantity of oil entering an engine at each stroke or revolution is very small, though in the aggregate it is considerable and with some lubricator very great. It does its work, however, by maintaining the lubrication of the cylinder and piston, and shows its presence and diffusion within the steam by reaching all parts of the inside of the engine, and if it is not separated, manifests itself most decidedly in the boiler, when the feed water is taken from a condenser or the returns of an exhaust steam heating system. This oil gathers in spots on the tubes or shell of the boiler, and prevents the rapid conduction of heat to the water. As a result the excessive heat, developed at these points, forms blisters by reducing the thickness of the metal and weakens the boiler. The oil in the exhaust steam, which is used for a heating system, collects on the radiating pipes and reduces the heat transmission. After these surfaces have been sufficiently coated, the oil is carried back to the boiler in the condensed steam if used for feed water. Much of the oil that passes through the engine is simply held in mechanical

which goes with the condensed steam within the steam pipe, and the second, the vapor or volatile form, which is carried entirely by the steam.

In a very large exhaust pipe, the draft, or mechanical force of the steam as it passes through the pipe is not sufficient to carry the water forward or upwards, and also takes place when the pipe is not performing its maximum duty. When the exhaust is small in diameter, and the force of the exhaust sharp, the water of condensation is carried along the exhaust pipe in a minute spray, and by the same action the oil that has passed through the engine, is carried along with the water and thrown forward in the direction that the steam is going, but at a much less velocity. The oil that remains vaporized, of course, will pass with the steam, no matter whether the velocity of the steam as it escapes through the exhaust pipe is high or low, as this oil is fixed within the steam and requires special treatment to separate it.

If the exhaust pipe is enlarged at some point until the steam as it passes through it has a velocity so low that it is not capable of carrying its own condensation or oil emulsion with it, we have one of the most important principles of the grease extractor. Again, by having the steam suddenly reversed or changed in direction in this enlargement, we have the main principle of separation. This separation is performed by centrifugal force, reverse current, or baffle plates.

In the separators employing centrifugal force the steam is given a rapid whirling motion, by a spiral, which throws the oil and water to the outside of the steam passage and flows to the collecting chamber below.

Reverse current separators cause the steam to change in direction, thereby throwing out the water and oil carried along with it, due to their momentum. The oil is collected in some suitable receptical below and the steam passes out above.

Baffle plate separators contains plates which are placed in the path of the steam, breaking up the current and changing its direction. The water and oil flow down the plates to the oil chamber below.

It is claimed by manufacturers of oil separators, and experience seems to show, that with the use of an efficient form of separator and with subsequent purification and settling of the feed water, it is safe to return condensation from the steam to the boilers as feed water. Instead of being used as feed water, this water can be used in shops for washing purposes and in laundrys for washing clothes. All oil separators have higher efficiencies at low velocities of the steam, due to carrying over of oil from splashing and agitation at the higher velocities.

The great difficulty in testing oil separators for efficiency is met with in determining the amount of oil in

the condensed steam discharged from the separator and condenser. There are but two methods known to be accurate for determining this percentage of oil.

The first method consists in taking a five-pound sample of the oil emulsion, from the separator or condenser, placing it in a large separatory funnel, adding ether and shaking up. The ether dissolves the oil out of the emulsion and floats on the water. This water is drawn off at the bottom of the funnel leaving the ether and oil. The latter solution is placed in a large flask, the ether evaporated by heating gently (usually by hot water). The ether vapor is condensed and may be used over again. The weight of oil remaining in the flask is easily determined, and knowing the original weight of the emulsion the percentage of oil is found.

In the second method a one-litre sample is used if the oil exceeds 0.01 gram per litre and 2 litres if less. If the oil exceeds 0.1 gram per litre a 500-c.c. sample is used, while if greater than 1 gram per litre only a 250-c.c. sample is necessary. Add to the sample contained in a 2 1/2 litre flask, about 5 c.c. of a "Ferric Chloride" solution and heat nearly to boiling; then add ammonia in excess to precipitate the iron (which precipitate contains the oil) and boil for at least 2 minutes. Allow it to stand a few minutes and filter through a 15 cm. fat-free, washed filter paper. The precipitate should be washed with hot water and thoroughly

dried on the filter paper. When dry the oil is extracted fr the precipitate with ether, in a Soxhlet apparatus, and when completed, the ether extract is evaporated leaving the oil in the flask, the weight of which can be easily determined by weighing.

The "Ferric Chloride" solution is made up as follows:- Dissolve 40 grams of ferric chloride in distilled water, and add 10 c.c. of hydrochloric and 1 c.c. of nitric acid, the whole being made up to 1 litre.

The first investigation into separating the oil from an oil emulsion was by an electrical process, based on the "Davis-Perrett" system of purifying condensed steam used for boiler feed. This system allows the oily water to flow between iron plates placed vertically in a tank 4 x 4 x 8 feet. The plates are connected alternately to the positive and negative poles of a direct current circuit, so that the current passes from one plate to the next across the flowing water. The action of the current is to cause the emulsified oil to coalesce thereby making it easy to filter out. By one theory the action is as follows:- the atoms of oil cling to the particles of the oxide of iron which come away from the plates owing to electrical action. Once a week the poles of the electric circuit are reversed to remove the oil clinging to the negative plate.

A method tried, similar to that of the Davis-Perrett,

for oil elimination was tested with a sample of oil emulsion obtained from the drip cock on an engine. The sample was placed in a glass jar 8 x 4 x 5 inches to a depth of 2 1/2 inches and two wrought iron plates, 7 1/2 x 2 x 1/16 inches, were immersed in it, 1/4 inch apart. Wires attached to the plates were connected, through a pole changer and lamp rack, to a 110 volt direct current lighting circuit. The current was raised to about one ampere at 50 volts by cutting in lamps on the rack and run for 5 minutes. By use of the pole changer, the direction of the current through the cell was changed once a minute, to remove the deposit on the negative plate. The oil coagulated leaving the water clear when filtered. The precipitate left on the filter paper was tested and found to contain iron while the filtrate showed no trace of it. If no iron had been present, the weight of the oil could be easily determined by knowing the weight of the filter paper. The iron plates were also used with alternating current at about 70 volts, 25 cycles, but the current flowing, even when the plates were but a sixteenth of an inch apart, was but a small fraction of an ampere which in ten minutes made no visible change in the emulsion. Evidently the reversals of the current were far to frequent to coagulate the oil.

Lead plates were used instead of the iron plates mentioned above and with eight lamps and the rack turned on, the plates were brought to within 1/64 of an inch from each

other before any visible action took place with about 1 ampere direct current at 75 volts. It seemed that the oil did not coagulate on reversing the current, but a fine lead oxide was given off quite freely. This lead was determined later by making a test as follows:- A test tube was partly filled with some of the solution, dilute nitric acid added in excess and boiled to dissolve any of the metal which was not in solution. Potassium bi-chromate was added giving a heavy gelatinous precipitate indicating lead. The electrolytic action on the plates was plainly visible showing that lead plates could not be used without obtaining lead in the coagulated oil.

Aluminum plates when used formed a precipitate very slowly with the plates close together and a current flowing of two amperes at 80 volts. The precipitate was very fine and gelatinous causing very slow filtration. The precipitate showed a slight trace of aluminum by boiling with nitric acid and adding ammonia. The filtrate gave no trace of aluminum. These plates showed the action of the current by becoming brighter and slightly roughened.

It was decided from these tests, that the coagulation of oil depended upon oxidation of the metal from electrolytic action and would make an ether extraction necessary to determine the amount of oil.

The Ferric-Chloride method was the next tried for the determination of the amount of oil in an emulsion. A sample

of condensed steam was taken from the exhaust of an engine, 1500 c.c. of which gave less than 0.1 gram of oil or .01%. The method used was as follows:- 1500 c.c. of the emulsion was heated almost to boiling in a 2 litre flask, after which 5 c.c. of a ferric-chloride solution was poured in, the mixture was boiled and ammonia to excess added, which precipitated, ferric-hydroxide and taking the oil along with it. By means of a siphon the water and precipitate in the flask, was drawn over into a 3 inch thimble filter set in a gooch funnel, the latter attached to a filter pump. After the solution was filtered, the thimble filter was dried and placed in a Soxhlet extractor, which consists of a cylindrical glass vessel and condenser attached above a small flask containing ether. By heating the flask the ether was evaporated, passing up a large glass tube, through the top of the extractor, to the condenser, where the ether was condensed, falling down on the thimble filter containing the precipitate. A small glass tube siphon connected to the bottom of the extractor and extending up along the outside, the height of the thimble filter, allowed sufficient ether to collect so as to cover the filter and to discharge back to the flask below. The ether dissolved the oil from the precipitate and deposited it in the flask. After ten similar extractions the flask was removed and weighed after drying. The flask was again weighed after removing the oil,

the difference between the two weights being the weight of oil.

Several different methods for filtering the precipitate of ferric hydroxide and oil, were tried. It was decided that by heating the two litre flask, containing the new formed precipitate in water and siphoning the contents into a 4 inch funnel, set in a one-litre filter bottle, and filtering through a 15 cm. fat-free, washed, filter paper, supported at the apex by a 3/4 inch platinum cone, that the operation was the shortest and most accurate. The filter bottle was attached to a compressed air filter pump so as to hasten the filtration, which lasted for about 30 minutes.

To facilitate the rapid testing of oil emulsions, permanent apparatus was built. A small cabinet was constructed having its inside dimensions 40 x 12 x 8 inches. This was divided into two sections, the lower being 10 x 8 x 12 inches and containing six 32 candle- power incandescent lamps set upright in three rows of two each. The central row is connected to the main circuit through a snap switch. The two outer rows are connected independently through snap switches to the main snap switch. This arrangement allows any combination of lights for heating purposes. All the sides and door of this section are lined with three thicknesses of 1/16 inch sheet asbestos, to prevent radiation. The upper section is large enough to hold two Soxhlet ex-

tractors and condensers. Two universal clamps support the apparatus above the lamps. The lips of the evaporating flasks rest on the partition and the flasks extend into the lower section where they are surrounded by four lamps each. Slots are cut in the partition from the front edge to the center to fit the necks of the flasks. The water connections to the condensers are rubber tubes which are led through the top of the cabinet.

In order to obtain more information on the formation of emulsions, live steam was blown through the high-pressure cylinder of a cross compound Corliss engine, having its valves removed. A lubricator was attached to the steam pipe just below the throttle to feed oil into the steam. The steam was by-passed around an oil separator and condensed. Samples of the condensed steam were taken, varying the velocities and oil supply. After 24 hours these samples were examined and the oil therein was found to be thoroughly emulsified. The formation of the emulsion was probably due to the high temperature and the violent agitation of the steam. This agitation was caused by the many turns of the steam current through the valves, cylinder, and exhaust piping. From the above results it was concluded that the velocity of the steam and quantity of oil supplied did not effect the emulsification of the oil. Again steam was passed through the engine, having the oil

separator by-passed but not supplying oil. After two hours, samples of the condensed steam were taken to give the lowest percentage of oil that was possible in the condensed steam.

The exhaust steam was passed through the oil separator and the lubricator started. This oil separator was of the ordinary baffle-plate type, having a small reservoir below. In order to take samples of emulsion from the separator when under a vacuum, an auxilliary reservoir made of a 3-inch pipe, 18 inches long and capped on both ends, was attached to the bottom of the separator, by a one inch pipe containing a globe valve. A drain pipe with a valve, a water glass, and an air cock were attached to this auxilliary reservoir. The discharge from the condenser was a thick emulsion, while no emulsion was removed by the separator. Upon the supposition that the steam passing through the separator was dry or superheated water was injected into the steam pipe by a small pump, so as to have the necessary wet steam for separation. After this the separator removed a thick oil emulsion and the condensed steam was clear.

Owing to the uncertainty of the quality of the steam passing through the separator, when injecting water into the live steam pipe, it was decided to run the engine when taking samples. With the corliss engine running, samples

of oil emulsion were taken from the separator and condensed steam at regular intervals for three hours.

The above samples of emulsions were thicker than usual due to oil being pumped into the high and low pressure cylinders and the lubricator being run at full capacity. This was done to obtain an emulsion which by various dilutions would give all the different grades usually taken from oil separators, or condensed steam under different conditions. Thus, four of the one-gallon samples were shaken up in a ten gallon demyou to be the original emulsion or standard sample. Two 500 gram samples were taken from this standard emulsion and tested both at the same time by the ferric-chloride method to determine the amount of oil in them. In these two tests the precipitates of ferric hydroxide and the oil were not dried but were inserted, while on the filter paper, into the thimble filter. The water in the heavy precipitate prevented the ether from taking out all the oil, the precipitate being oily to touch, and while some of it was carried with the ether into the evaporating flask. This water floated on the oil in the flask, and was removed by drying for two days in a dessicator. Two other samples were taken from the original emulsion and tested for the percentage of oil contained in each. The precipitates, obtained from these samples, were dried thoroughly and washed with ether, the oil and ether being collected in the evaporating flask before

being inserted in the thimble filters. Then after ten ether extractions in the Soxhlet apparatus, the precipitate was a dry fine powder, showing that all the oil had been removed. An average of the percentages of oil contained in the two samples, was taken as the correct percentage in the standard emulsion.

The amount of oil being determined in the standard emulsion, a plan was laid out for dilutions to obtain a set of samples varying in color or percent of oil, from the original to a faint milky color. Thus by diluting parts of the standard emulsion in a graduate the change in color was noted and 24 samples were determined, upon which would give a gradual change between these limits. Each sample was made up as follows:- A small amount of the original emulsion was carefully weighed in a beaker, and distilled water added to make up the required dilution by weight. These samples were put up in 250 c.c. oil sample bottles, and labeled with the number and percentage of oil.

CALCULATIONS.

Percentage of oil in original emulsion:-

#1.

Weight of flask and emulsion------------------609.2 grs.
Weight of flask,------------------------------110.0 grs.
Weight of emulsion,---------------------------499.2 grs.

Weight of evaporating flask and oil-----------31.5764 grs.
Weight of evaporating flask,------------------29.9307 grs.
Weight of oil,-------------------------------- 1.6457 grs.

Percentage of oil, $\frac{1.6457}{499.2} = 0.3297\%$.

#2.

Weight of flask and emulsion,-----------------433.8 grs.
Weight of flask,------------------------------109.8 grs.
Weight of emulsion----------------------------324.0 grs.

Weight of evaporating flask and oil,----------30.9756 grs.
Weight of evaporating flask,------------------29.9281 grs.
Weight of oil,-------------------------------- 1.0475 grs.

Percentage of oil,= $\frac{1.0475}{324.0} = 0.3233\%$

Dilution of Original Emulsion:-

No.	Dilutions	% Oil.	No.	Dilutions	% Oil.
1	0	0.3233	13	45	0.00703
2	1/2	0.2154	14	55	0.00577
3	1	0.1616	15	65	0.00490
4	2	0.1077	16	75	0.00425
5	3	0.0808	17	85	0.00376
6	5	0.0538	18	100	0.00323
7	7	0.0404	19	115	0.00279
8	10	0.0323	20	130	0.00246
9	13	0.0231	21	150	0.00214
10	18	0.0170	22	175	0.00184
11	25	0.0124	23	200	0.00161
12	35	0.00897	24	300	0.00107

Calculations for sample No. 18; - 100 Dilutions.

Weight of beaker and emulsion,------------61.013 grs.

Weight of beaker,--------------------------58.620 grs.

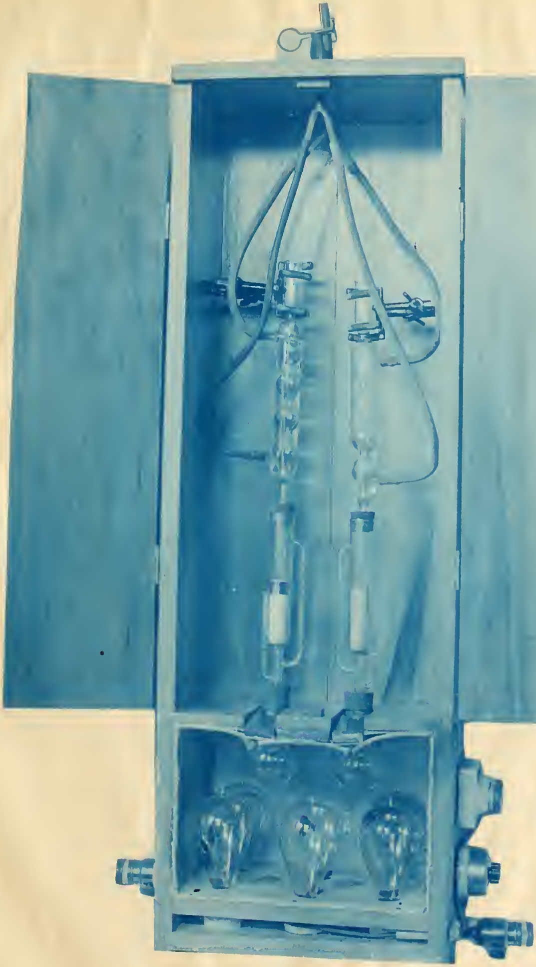
Weight of emulsion,---------------------- 2.393 grs.

x 100

Weight of distilled water,-------------- 239.3 grs.

Weight of beaker and emulsion,------------ 61.013 grs.

Total weight on balance------------------- 300.313 grs.



CONCLUSION.

The ferric-chloride method finally determined upon, is briefly as follows:-

Quantity of sample to be used:-

If the oil is less than 0.01 gram per litre:- use 2 litres.
" " " " greater than 0.01 gram per litre:- use 1 litre.
" " " " " " 0.1 " " " :- " 500 c.c.
" " " " " " 1.0 " " " :- " 250 c.c.

Add to the sample contained in a 2 1/2 litre flask, about 5c.c. of a "Ferric Chloride solution and heat nearly to boiling; then add ammonia in excess to precipitate the iron, and boil for at least two minutes. Siphon the precipitate and solution on to a 15 cm., fat-free filter paper. Wash the precipitate adhereing to the inside of the flask on the filter paper with hot distilled water. This filter paper is held in a four-inch, 60-degree funnel and supported at its tip by a 3/4 inch, perforated platinum cone. The glass funnel is supported in a one litre filter flask which is connected to a filter pump. The filter paper and precipitate are thoroughly dried and cooled. While the filter paper is on the funnel wash the precipitate with ether into the evaporating flask of a Soxhlet extractor. Practically all the oil is dissolved, and carried into the flask by the ether. The filter paper with its precipitate, is inserted into a fat-free, three inch thimble filter, which is set in a Soxhlet extractor.

Fill the evaporating flask with more than enough ether to start the siphon of the extractor. Heat the evaporating flask, while on the extractor, until at least ten extractions have been made. The thimble filter is then removed and the ether evaporated from the oil, being collected in the extractor. The flask and oil is thoroughly dried in a desicator and then weighed on an accurate balance. After cleaning the flask thoroughly with water and ether, and drying in a dessicator, it is again weighed. This weight subtracted from the first gives the weight of oil in the emulsion.

To make the "Ferric Chloride" solution dissolve, 40 grams of ferricchloride in distilled water and add 10 c.c. of Hydrochloric and one c.c. of nitric acid, the whole being made up to one litre. The results obtained from careful tests performed according to the above directions should not show a variation of more than 2%.

By comparing any emulsion, obtained from a separator or condensed steam, with the 24 standard samples, the percentage of oil can be easily determined by comparison. The errors in this method are negligable for the maximum error can be no more than one-half the difference between any two of the standard samples.

OIL SEPARATORS.

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